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## THE MECHANISM OF GLASS POLISHING<sup>1</sup>

By J. G. SMITH AND J. G. HOOLEY

### ABSTRACT

In an effort to determine whether glass flows when polished, uranium glasses were fused to nonuranium glasses and polished in a direction from the active to the inactive side. Autoradiographs of alpha tracks were taken before and after to detect any transfer of glass. The method would detect a uniformly flowed layer 4 Å thick if it were over a distance greater than 0.2 mm. No evidence for such a flow was found in the case of a typical silicate or phosphate glass polished with a water slurry of ceric oxide or rouge. Radioactive ceric oxide was also used to polish nonactive glass. If the contact temperature approached the softening point of the glass and caused flow, then some of the ceric oxide might become incorporated in the surface. The amount was less than  $4 \times 10^{-8}$  gm. per sq. cm.

### INTRODUCTION

Before glass is polished, the surface is ground to the desired contour with an abrasive slurried with water on a revolving iron form. This process is recognized to be one of chipping particles of glass away from the surface and leaves the latter rough and translucent.

In the ensuing polishing, a finer powder is used, slurried with water on moving felt or wax. The powder particles must be hard and insoluble and have a melting point higher than the softening point of the glass being polished. Rouge and ceric oxide are in common use today and were used in this work.

Previous work (2) indicates that one or more of three processes are active in the polishing operation. The first of these is simply a continuation of the grinding operation, a chipping off process, carried to such a degree of fineness that the reflected light waves show no measurable amount of straying. In the second, the frictional heat of polishing raises the surface temperature to the softening point of the glass and thereby smooths the surface by an actual flow of glass. The third is chemical attack of the surface by the liquid in which the polishing powder is suspended.

In support of polishing by removal of glass, Preston (7) found no change in the rate of removal of glass as polishing progressed. This, he reasoned, was incompatible with the existence of a flowed layer which would presumably be of a different structure and therefore removed at a different rate. On polished crystals such a layer exists and does change the rate of polishing.

<sup>1</sup> Manuscript received August 19, 1952.

Contribution from Department of Chemistry, University of British Columbia, Vancouver, B.C.

In support of a flow of glass during polishing, various workers (4, 6, 8) find that a surface polished and then etched exhibits the original milling pattern acquired during the rough grinding. They believe that glass from peaks has flowed into valleys and is more easily removed by the etching solution.

Chemical attack by water has also been suggested as playing a part in polishing (1, 5). For instance a silica gel might form and be removed by the polisher wherever it projects above the common level (5).

The purpose of our work was to detect any flow of glass during polishing. Two approaches were used. In the one radioactive glass was fused to nonactive glass, ground, and then polished in such a direction that any flow would transfer active glass to nonactive. Autoradiographs were used for detection of such a transfer. In the other approach, glass was polished with radioactive ceric oxide powder. If flow occurred, then the surface temperature must have approached the softening point of the glass and might have caused incorporation of the active powder with the glass. Autoradiographs and a Geiger counter were used for detection.

#### EXPERIMENTAL DETAILS AND RESULTS

##### *First Approach*

The sensitivity of the autoradiographic technique was first determined by exposing an Eastman NTB-3 plate to a uniform layer of uranium nitrate of known thickness. An area of 8.85 cm.<sup>2</sup> was covered with  $3.07 \times 10^{-5}$  gm. of uranyl nitrate hexahydrate, using the method of Cook and Hudswell (3). A plate was placed in contact with this for two hours and developed. The alpha track density was determined visually under a magnification of 560 and a cross hatched field of known dimensions. It appeared to be uniform and gave the densities listed in Table I in three different areas. The results for seven

TABLE I  
CALIBRATION OF ALPHA SENSITIVE PLATES

Sample	Area, cm. <sup>2</sup>	Tracks	Density, cm. <sup>-2</sup>
Standard	0.410	114	278
"	0.454	128	282
"	0.404	111	274
Totals	1.268	353	Av.* 278 ± 15
Blank	0.423	59	139
"	0.388	53	136
"	0.377	52	138
"	0.325	42	129
"	0.390	54	138
"	0.372	51	137
"	0.342	44	129
Totals	2.617	355	Av.* 136 ± 7

\*Calculated from the totals.

NOTE: Corrected density of standard =  $142 \pm 17$  cm.<sup>-2</sup>.



blanks for as many different plates are also given in Table I. They show the expected or better reproducibility and a density of  $142 \pm 17$  track  $\text{cm}^{-2}$  from two hours contact with  $3.47 \times 10^{-6}$  gm.  $\text{cm}^{-2}$  of uranyl nitrate. Calculation shows that for a 100 hr. exposure to give a track density of 20  $\text{cm}^{-2}$  above background,  $10^{-8}$  gm.  $\text{cm}^{-2}$  of the nitrate would be required. This is the amount of uranium in 1 sq. cm. of glass 4 Å thick and containing 5%  $\text{U}_3\text{O}_8$ . Such a layer is considered to be our lower limit of sensitivity.

Slabs of silicate and phosphate glass were then prepared both with 5%  $\text{U}_3\text{O}_8$  and with no added activity. The compositions were:

$\text{SiO}_2$	56%	$\text{P}_2\text{O}_5$	67%
$\text{Na}_2\text{O}$	14%	$\text{Al}_2\text{O}_3$	4%
$\text{PbO}$	30%	$\text{CaO}$	10%
		$\text{BaO}$	19%

For each composition, the active and inactive slabs were sealed together along an edge and ground to a plane surface in a direction from the inactive to the active side. An autoradiograph taken at this point showed only the background count on the inactive side. The slab was then polished with a revolving felt-covered disk steadily supplied with a water slurry of rouge or ceric oxide and exerting a pressure of 300 gm. per sq. cm. on the 6 sq. cm. sample. The direction of motion was from the active to the inactive side. A second autoradiograph was taken to show any flow. The results for the following variations in conditions were:

#### *Time, Glass, and Polishing Powder*

The silicate and phosphate glasses were each polished with ceric oxide and with rouge. A 0.5  $\text{cm}^2$  area 0.2 cm. from the boundary was counted at various times during the operation. The results in Table II show that there was no flow of active glass to this area at any time during the polishing.

TABLE II  
TRACK DENSITIES DURING POLISHING

Time, sec.	Tracks, $\text{cm}^{-2}$	Time, sec.	Tracks, $\text{cm}^{-2}$
Ceric oxide on silicate		Ceric oxide on phosphate	
0	136	0	136
5	130	5	133
20	128	15	138
60	132	50	135
240	136	120	130
480	132	240	129
600	132		
Rouge on silicate		Rouge on phosphate	
5	129	5	137
20	136	15	130
50	135	50	141
100	133	120	136
200	136	240	129

### *Proximity to the Boundary*

The above results show no flow to an area 0.2 cm. from the boundary between active and inactive glass. For detection of flow over a shorter distance, a narrow strip about 0.01 cm. wide and only 0.02 cm. from the sharp boundary was counted on each of 18 plates. These were all of silicate glass polished with ceric oxide. The total area of 0.5 sq. cm. contained 138 tracks per sq. cm. Again there is no evidence of flow this time over 0.02 cm.

### *Dry Polishing*

Silicate and phosphate glasses were polished with rouge and ceric oxide for 20 min. wet and then 10 min. dry. The results in Table III are for an area 0.2 cm. from the boundary. They show no flow of active glass to this area even

TABLE III  
TRACK DENSITIES FOR A DRY POLISH

Glass	Powder	Tracks, cm. <sup>-2</sup>
Silicate	Ceric oxide	133
Phosphate	Ceric oxide	129
Silicate	Rouge	131
Phosphate	Rouge	129
Blank		136

at the higher temperature developed during the dry period. Counts made 0.2 mm. from the boundary gave a density there of  $141 \pm 37$  cm.<sup>-2</sup>. The larger uncertainty is the result of having only three plates available.

### *Second Approach*

The above results show no flow of glass greater than about 4 Å thick over a distance greater than 0.02 cm. It does not, however, preclude the flow of glass from a peak into an adjoining valley or fissure. If this did occur, some of the polishing agent might become incorporated into the glass by fusion with it. To detect this, neutron activated ceric oxide polishing powder was used. It had a half life of 30 days and a specific activity of 0.2 mc. per gm.

Both silicate and phosphate glasses were polished with this powder. During the early stages of polishing a few hundred counts per minute were always found on about 6 sq. cm. of surface. They were not removable by washing and scrubbing but were removed in a few seconds by warm 6*N* hydrochloric acid containing potassium iodide. An autoradiograph showed that the activity was concentrated in minute pits and scratches just visible to the unaided eye. As these were removed by further polishing the count decreased. We were able to get it down to  $4 \pm 1$  per min. per sq. cm. and no darkening on the autoradiograph. This corresponds to  $4 \times 10^{-8}$  gm. of ceric oxide per sq. cm. Such an amount would be present in a surface layer 30 Å thick and containing 5% ceric oxide. This is so close to the lower limit of detectability that the existence



of such a layer is not considered to be proved by these experiments. Certainly no greater amount is incorporated and the minute amount under question might be trapped in invisible pits or scratches.

#### CONCLUSION

The general conclusion is that the polishing of glass involves at least primarily a simple removal of material and not a flow.

#### ACKNOWLEDGMENT

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# A PILOT PLANT EVAPORATOR AND DISTILLATION UNIT<sup>1</sup>

By J. A. WHEAT<sup>2</sup>

## ABSTRACT

An evaporator with a heat transfer area of 26.2 sq. ft. and a packed distillation column 4 in. in diameter are described. Auxiliary equipment included a reboiler for the column, an entrainment separator, a condenser, feed and product tanks, and pumps. All units were fabricated of A.I.S.I. No. 316 stainless steel. Continuous operation at steady conditions was attained by extensive use of automatic control instruments. The equipment was used to evaporate fermented molasses mash in single stage operation and also in three stage operation designed to simulate triple effect evaporation. It was also used for the recovery and purification of 2,3-butanediol from an aqueous solution obtained after separating diol from fermented mash solids.

## INTRODUCTION

In pilot plant investigations of the production of 2,3-butanediol by fermentation, the need arose for a small evaporator and distillation column for recovery of diol from aqueous solutions. Equipment used for the purification of diol recovered from whole wheat mash (6) was unsatisfactory in several respects. The copper components were subject to corrosion by ammoniacal solutions, several desirable process steps could not be performed, and operation was difficult. The unit was therefore replaced by equipment designed for ease and flexibility in its operation. The equipment and piping were made of A.I.S.I. No. 316 stainless steel to give corrosion resistance to a wide range of chemicals. The unit was designed to operate at pressures from full vacuum to 50 p.s.i. gauge. Indicating, recording, and controlling instruments were installed to aid in the operation of the unit and to provide temperature measurements at a large number of points.

A description of the equipment and instrumentation is followed by results of performance tests. Data are then given for several process steps with fermented mash and recovered diol solutions.

## EQUIPMENT

Fig. 1 is a photograph of the equipment on the operational floor. At the left is the top portion of the evaporator and to the right of it is the reboiler and bottom of the distillation column. The bottoms receiver is below the reboiler and the distillate receivers are behind it, next to the wall. Fig. 2 is a piping diagram of the equipment and instruments.

### *Equipment Components*

The evaporator was patterned after the long-tube, natural-circulation type (4) but there was only sufficient head room to allow tubes 6 ft. long.

<sup>1</sup> Manuscript received July 31, 1952, and, as revised, December 5, 1952.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa, Canada. Issued as Paper No. 147 on the Uses of Plant Products, and as N.R.C. No. 2917.

<sup>2</sup> Chemical Engineer, Industrial Utilization Investigations.

Nineteen 1-in. tubes, giving a heat transfer area on the liquid side of 26.2 sq. ft., were installed in a steam chest 8 in. in diameter, surmounted by a vapor head 14 in. in diameter and approximately 24 in. high. A gauge glass ran almost the full height of the evaporator from below the tubes to the middle

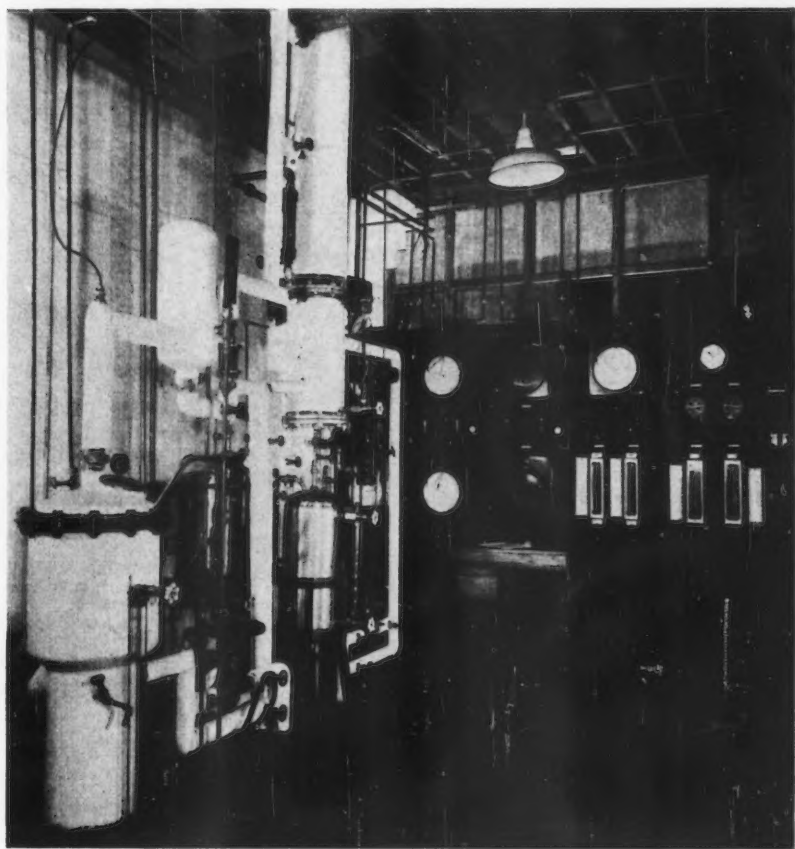


FIG. 1. Photograph of the equipment on the operational floor.

of the vapor head. Two sight glasses 4 in. in diameter were installed in the vapor head, one on the top and one on the side. A baffle was placed over the top of the tubes to reduce entrainment.

The distillation column, 4 in. by 12 ft., was packed to a height of 10.33 ft. with  $\frac{1}{4}$  in. Raschig rings. The packing was supported on a conical plate drilled with 200 holes  $\frac{3}{16}$  in. in diameter, giving a free area of 44%. A 3 in. sight glass was installed two-thirds of the way up the column. Feed could be added below the packing support plate or at five optional points at 2-ft.

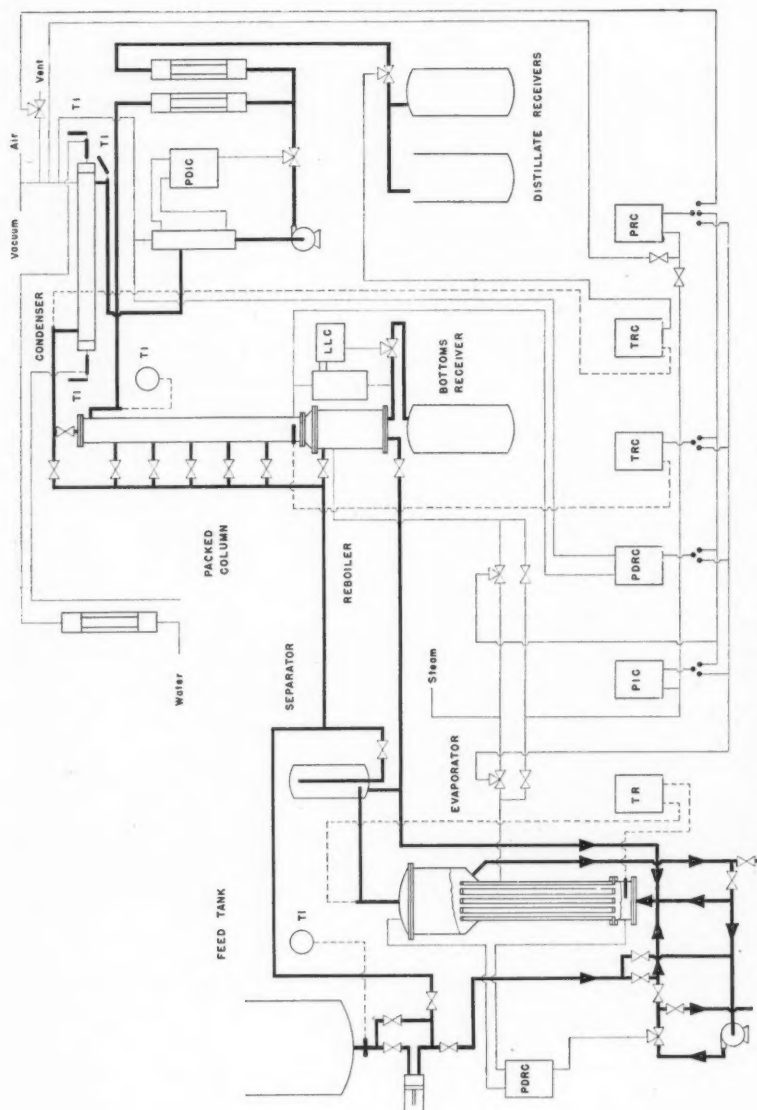


FIG. 2. Piping diagram of the equipment.  $T$  = temperature,  $P$  = pressure,  $PD$  = pressure difference,  $LL$  = liquid level,  $R$  = recorder,  $I$  = indicator,  $C$  = controller.

intervals up the column. Reflux was admitted through a  $\frac{1}{2}$  in. pipe and released at the center of the column but no special distributor was provided. The column was insulated with asbestos aircell 4 in. thick. The column reboiler was 25 in. long by 8 in. in diameter and was fitted with a gauge glass. The heating element, which was 6 in. in diameter, contained 37 tubes  $\frac{1}{2}$  in. O.D. by 15 in. long, giving a liquid-side heat transfer area of 5.65 sq. ft.

An entrainment separator, 6 in. in diameter by 18 in. high, was placed between the evaporator and column. Vapor entered tangentially at the bottom and was removed through a 1 in. pipe entering the bottom and extending almost to the top. The condenser contained 10 tubes  $\frac{1}{2}$  in. O.D. by 6 ft. long, installed inside a length of 2.5 in. pipe, fitted at the ends with caps which were drilled and tapped for water lines. The outside heat transfer area was 6.54 sq. ft. The feed tank had a capacity of 50 gal. and each of the three product receivers, 10 gal. The bottoms receiver and one of the distillate receivers were of the closed type; the other two tanks were open.

#### *Pumps and Piping*

Feed was pumped to the evaporator or to the column by a piston pump with a capacity of 5 gal. per hr. A small, two-stage, centrifugal pump with a capacity of 2 gal. per min. at 25 p.s.i. was installed at the bottom of the evaporator to add feed to the evaporator or to discharge concentrate. The arrows at the base of the evaporator in Fig. 2 indicate the flow for continuous operation, with feed controlled by the piston pump, and draw-off sent through the control valve by the centrifugal pump. Condensate was pumped with a single-stage centrifugal pump through reflux and take-off rotameters. To prevent the pump from running dry and to give a smooth discharge rate, a constant liquid level was maintained in a condensate receiver made of a 2.5 ft. length of 3 in. pipe. Liquid from the base of the column could flow to the bottoms receiver or be returned to the bottom of the evaporator along with the liquid collected in the entrainment separator.

#### *Instrumentation*

Liquid level in the evaporator was measured with a mercury manometer type of instrument using an air purge. This instrument was used to control either feed or discharge by reversing the action of the controller and by using the appropriate flow arrangement. Liquid level in the condensate receiver was measured in the same manner. Liquid level in the reboiler was controlled with a nonindicating instrument of the displacement type.

Instruments indicated at the bottom of Fig. 2 will be described in the order shown. The first (TR) was a two-pen recorder to measure liquid and vapor temperatures in the evaporator. The second (PIC) was an indicating pressure controller for measuring and controlling the steam pressure in the heating element of either the evaporator or the reboiler. The third (PDRC), a mercury manometer with an air purge, was used to record the pressure drop across the column and to regulate steam to either the evaporator or the reboiler. The next two instruments (TRC) were in the same case but, for clarity, are shown

separately in Fig. 2. They measured and recorded the temperatures at the top and bottom of the column. The bottom temperature could be used to control either of the steam flows and the top temperature to control the take-off rate. The last instrument (PRC) on Fig. 2 was a compound pressure recorder-controller with a range of 30 in. vacuum to 65 p.s.i. gauge, and was used to record and control the pressure in the column. For pressures under atmospheric, the controller regulated the air allowed into the system; for pressures above atmospheric, compressed air was supplied to the column and the controller regulated the air vented. When the unit was operated at atmospheric pressure the instrument was available for recording and controlling either one of the steam pressures. The air output of the instruments was switched from one valve to another with air switches placed on the instrument panel. These switches and the bubblers used to regulate air purges may be seen in Fig. 1.

Air purging effectively prevented liquid from reaching the instruments from the top and bottom of the evaporator and the bottom of the column. However, some difficulty was caused by liquid entering the pipes leading from the upper floor to the instruments and suitable drainage chambers had to be provided. Liquid entered these pipes principally during starting-up operations and during admission of air after operation at reduced pressure.

Dial type thermometers were used to indicate the temperature of the feed and the reflux to the column. Industrial mercury-in-glass thermometers were placed in the condenser condensate and in the water to and from the condenser. The water rate to the condenser was measured with a rotameter.

#### EQUIPMENT TESTS

##### *Heat Losses*

The heat loss from the steam chest of the evaporator was determined by weighing the condensate formed when steam was turned on with no liquid in the evaporator. Heat loss could be expressed as:

$$L = 54.6P + 4082$$

where  $L$  is the heat loss in B.t.u. per hr. and  $P$  is the steam pressure in p.s.i. gauge.

Two tests were made with water to determine directly the heat loss from the column and from the entrainment separator at atmospheric pressure. Water was fed to the evaporator with the centrifugal pump at a rate controlled by the evaporator liquid level. Steam to the evaporator was controlled by the column pressure drop. No reflux was returned to the column, and the condensate formed by heat loss was collected at the base of the column and the separator. The average heat loss from the separator was 1130 B.t.u. per hr. and from the column, 3950 B.t.u. per hr. The calculated heat loss from a 4 in. column through four inches of asbestos air cell is only 400 B.t.u. per hr. The high experimental loss is due to added losses from exposed flanges and connecting pipe. Therefore thicker insulation on the column would not reduce heat loss appreciably.



In the two tests described above, the heat loss from the vapor head of the evaporator amounted to 38,000 B.t.u. per hr. In process tests, however, the loss ranged from 7000 to 40,000 B.t.u. per hr. There is no obvious explanation for the large variation during process tests.

### Heat Transfer to Water

Data on the transfer of heat to boiling water were obtained by operating the unit at total reflux with no feed or discharge. The pressure recorder controller was used to maintain a constant steam pressure, and temperature differences were calculated from the steam pressure and temperature of the vapor leaving the evaporator. The heat transferred was determined by weighing the steam condensate over a timed interval and correcting for heat loss from the steam chest and for flash evaporation of the condensate. Results obtained after cleaning the tubes with a rotating wire brush are plotted in Fig. 3 as a

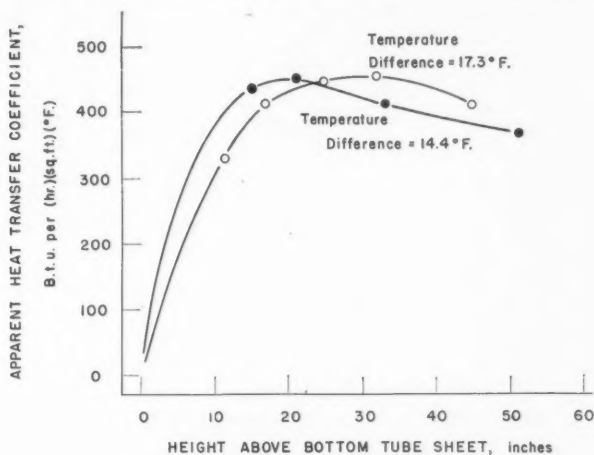


FIG. 3. Apparent heat transfer coefficient to boiling water in the evaporator as a function of liquid level.

function of liquid level for two temperature differences. The height of liquid above the bottom tube sheet was determined by correcting the reading of the liquid level instrument for density and for the relative location of the instrument tap and the tube sheet. The general slope of the curves is similar to those published by Perry (4, p. 502). The coefficient decreased at low liquid levels because the upper portion of the tubes was dry. At low liquid levels a lower coefficient was obtained at the higher temperature difference because a larger portion of the tubes was dry. At levels of 30 in. and above, liquid was ejected from the top of the tubes and the higher temperature difference gave higher coefficients.

Linden and Montillon (3; 4, p. 503) report a coefficient of 700 B.t.u. per (hr.) (sq. ft.) (°F.) at a temperature difference of 17° F. for an inclined, clean,

copper tube, 1 in. iron pipe size by 4.08 ft. long, which, except for the material and surface condition, would be expected to give results comparable to those obtained here. For the stainless steel evaporator being described, it was calculated that, at an over-all coefficient of 400 B.t.u. per (hr.) (sq. ft.) ( $^{\circ}$  F.), the resistance of the tube wall was 23.4% of the total. With the same film coefficients, the over-all coefficient would be 522 if the tube wall offered no resistance to heat transfer and 515 if the tube was copper. Surface conditions could easily change the coefficient by 200 units (4, p. 505). Values similar to those given in Fig. 3 have been obtained in a stainless steel evaporator comparable to the one described here (5).

Heat transfer coefficients to condensing water vapor in the condenser are given in Table I. These data were obtained under conditions which gave condensates very close to the boiling point. During process tests, however, the condenser was operated to give a condensate at a temperature considerably below the boiling point in order to reduce material losses by evaporation. The transfer coefficients of Table I give a smooth curve when plotted against water rate.

TABLE I  
CONDENSER HEAT TRANSFER COEFFICIENTS

Water rate, gm. per min.	Temp. diff., $^{\circ}$ F.	Coefficient, B.t.u. per (hr.) (sq. ft.) ( $^{\circ}$ F.)
0.42	64	78
0.44	71	76
0.46	73	78
0.59	74	95
0.75	66	121
1.41	85	180
1.44	67	183

### Column Capacity

In Fig. 4, the pressure drop across the column at total reflux is plotted against vapor rate. The experimental data were determined by operating the column at controlled pressure drops and observing the flow rate through the previously calibrated reflux rotameter. The vapor rate at the top of the packing was obtained by adding the amount condensed by cold reflux and, to obtain the average rate in the column, one-half the amount condensed by heat loss. A calculated pressure drop curve is also given in Fig. 4. The pressure drop for dry packing was calculated by the method of Leva and Grummer (2) using their equation for clay, Alundum, and other similarly rough materials. This value was multiplied by 1.5 (4, p. 395) to allow for the liquid flow. The resulting expression for dumped and pounded  $\frac{1}{4}$  in. Raschig rings is

$$P.D. = 0.00319 W^{1.9}$$

where *P.D.* is the pressure drop in inches of water per foot of packing and *W* is the flow rate in lb. per hr.

The flooding rate for operation at total reflux was estimated from two sets of data (4, p. 684) to be 50.5 and 36.1 lb. per hr. The experimental curve in Fig. 4 indicates that the flooding rate is slightly greater than 50 lb. per hr. At less than total reflux, the flooding rate would be higher.

The pressure drop between the evaporator and column was found to be about 10% of the column pressure drop at both atmospheric pressure and 26 in. of mercury vacuum.

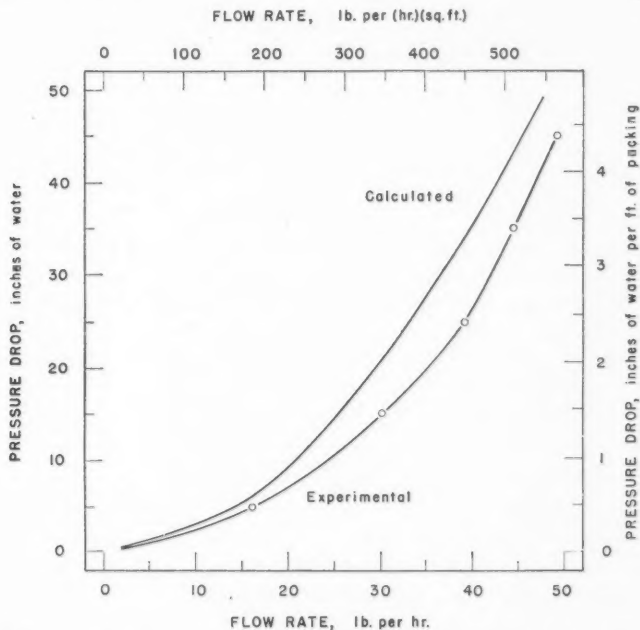


FIG. 4. Pressure drop across the column when operated with water at total reflux and atmospheric pressure.

#### Column Performance

Mass transfer data for the column were determined at atmospheric pressure with the system ethanol-water. Using the evaporator as a still, the column was operated at total reflux at controlled pressure drops. The amount and concentration of the charge was chosen to give ethanol concentrations at the bottom of the column of about 4 mole % and 83 mole % at the top. The unit was operated for an hour or more before readings and samples were taken. Flowmeter readings were corrected for differences in specific gravity and the amount condensed by cold reflux was added to obtain the flow rate at the top of the packing. Samples were taken by slowly bleeding off small portions of the reflux and the liquid leaving the bottom of the column. The ethanol content of these samples was determined with a hydrometer. The number of

over-all gas-phase transfer units was calculated (4, p. 552) using the vapor-liquid equilibrium data given by Perry (4, p. 574). Transfer unit heights are given in Table II. Since the results did not vary with vapor rate they were averaged to give a value of 15.3 in.

TABLE II  
MASS TRANSFER UNIT HEIGHTS FOR THE SYSTEM ETHANOL-WATER

Column pressure drop, in. of water	Flow rate, lb. moles per (hr.) (sq. ft.)	Transfer unit height, in.
5	3.74	13.8
15	7.60	18.3
25	9.43	15.4
35	10.61	13.6
45	11.40	14.9
Average		15.3

Transfer unit heights were also determined for the system 2,3-butanediol-water at reduced pressure. At the end of two process tests the column was operated at total reflux using the reboiler as a still. After steady conditions had been attained, samples were taken from the reboiler and from the reflux and analyzed for butanediol. Transfer unit heights were calculated as before using the vapor-liquid equilibrium data of Blom *et al.* (1).

Results of the two tests were:

Pressure, mm. of Hg	147	173
Flow rate at top of column, lb. per hr.	5.4	6.2
Concentration at top of column, mole %	0.291	0.076
Concentration at bottom of column, mole %	99.5	90.9
Transfer unit height, in.	19.4	15.4

## PROCESS TESTS

### EVAPORATION OF FERMENTED MASH

#### *Single-stage Evaporation*

Several batches of fermented molasses mashes were evaporated in the unit. The purpose of these runs was not to process pilot plant mashes but to obtain data for continuous evaporation of fermented mash. The capacity of the unit was too small for processing purposes and the larger evaporator in the pilot plant (6) was too large to allow runs of suitable length to obtain data on continuous operation.

To start the unit, the evaporator was charged with 6-7 gal. of feed. Steam was controlled to give the desired pressure drop and the take-off was manually regulated to give a reflux ratio (reflux/take-off) of about 1.0. Feed was added with the centrifugal pump and was controlled by the liquid level. When it was estimated by a material balance that the material in the evaporator had been concentrated to the desired point, the method of operation was changed to allow withdrawal of syrup from the evaporator. Feed was then added with

the piston pump at a constant and known rate. Distillate take-off was regulated manually at the correct proportion of the feed rate and syrup was pumped out with the centrifugal pump at a rate controlled by the evaporator liquid level. Each 10 gal. of distillate was sampled and discarded. Syrup was collected in an open tub, weighed every half hour and, at the end of each eight hour shift, sampled and emptied into a storage tank. Every two hours during a run, the steam flow was determined by weighing condensate and all indicated temperatures were noted.

Table III presents a material balance for 56 hr. of continuous operation. During the test period, the average vapor rate in the column was 30.0 lb. per hr. at the top and 34.1 at the bottom; the respective liquid rates were 7.4 and 11.5 lb. per hr.; and the average feed rate was 25.30 lb. per hr.

TABLE III  
MATERIAL BALANCE FOR SINGLE STAGE EVAPORATION  
OF FERMENTED MASH  
(Period of operation: 56 hr.)

	Total material	Diol	Solids
Feed, lb.	1416.5	29.89	46.46
Distillate, lb.	1258.7	3.03	0.00
Syrup, lb.	152.5	27.69	47.56
Out, lb.	1411.2	30.72	47.56
Out, % of feed	99.63	102.78	102.37
Loss, lb.	5.3	-0.83	-1.10

One single stage evaporation using once-through operation was carried out but, because of the large portion of the feed being evaporated, it was not possible to maintain steady conditions. As shown in Table III, the syrup amounted to only slightly more than 10% of the feed. Hence a slight variation in steam flow, although making only a small change in the distillate rate, had a pronounced effect on the syrup rate.

#### Three-stage Evaporation

To simulate triple effect evaporation, one batch of fermented mash was evaporated in three stages. The concentrated material of one stage was stored and used as feed for the next stage. The conditions used for the three stages were:

Stage	I	II	III
Effect	2	3	1
Pressure, p.s.i. gauge	12	atm.	25
Temperature, top of column, ° F.	242.6	212.0	266.0
Liquid/vapor, top of column	0.477	0.296	0.319
Steam pressure, p.s.i.	18.2	4.2	38.3
Evaporator vapor temperature, ° F.	243.9	214.0	270.3
Temperature difference, ° F.	11.8	10.9	14.4
Heat transfer coefficient, B.t.u. per (hr.) (sq. ft.) (° F.)	243	214	237
Evaporator liquid level, inches above bottom tube sheet	46	45	43

Material balances over periods of continuous operation are given in Table IV.

TABLE IV

MATERIAL BALANCES FOR THREE STAGE EVAPORATION OF FERMENTED MASH  
(Periods of operation: Stage I—72 hr.; Stage II—72 hr.; Stage III—40 hr.)

Stage	I		II		III	
	Total material	Diol	Total material	Diol	Total material	Diol
Feed, lb.	3745.5	99.63	3131.3	116.95	1480.1	93.75
Distillate, lb.	935.5	0.29	1228.4	0.07	763.3	0.24
Syrup, lb.	2688.2	102.69	1828.4	113.45	631.5	93.84
Out, lb.	3623.7	102.98	3056.8	113.52	1394.8	94.08
Out, % of feed	96.75	103.30	97.62	97.07	94.24	100.35
Loss, lb.	121.8	-3.36	74.5	3.43	85.3	-0.33

### Heat Transfer

Two tests were made to determine the extent of fouling of the heat transfer surface by concentrated fermented mash. Syrup was boiled at total reflux and steam flow measurements were taken at various times. Temperature differences and liquid levels were determined as described previously for heat transfer coefficients to water. The vapor temperature includes the boiling point elevation caused by butanediol but not the elevation caused by dissolved solids. In the first test, the solids concentration was 38.2%. An initial heat transfer coefficient of 113 B.t.u. per (hr.) (sq. ft.) ( $^{\circ}$ F.) decreased to 66 in 96 hr. Since the heat flow was constant, the temperature difference increased from 29 $^{\circ}$ F. to 47 $^{\circ}$ F. In the second test, at a solids concentration of 62.9%, the initial coefficient of 137 B.t.u. per (hr.) (sq. ft.) ( $^{\circ}$ F.) decreased to 41 in 72 hr., the temperature difference increasing from 27 $^{\circ}$ F. to 58 $^{\circ}$ F. These tests indicated that fouling occurred rapidly. This rapid fouling possibly explains why

TABLE V

EVAPORATOR HEAT TRANSFER COEFFICIENTS TO BOILING SYRUP  
DURING ONE PROCESS TEST

(Evaporator liquid level: 30 in. above bottom tube sheet)

Shift	Solids in syrup, %	Temp. diff., $^{\circ}$ F.	Coefficient, B.t.u. per (hr.) (sq. ft.) ( $^{\circ}$ F.)
1	32.6	8.0	236
2	30.6	7.5	252
3	28.9	9.5	212
4	32.8	10.7	186
5	30.8	9.1	213
6	31.2	10.7	191
7	32.8	10.6	196
8	31.6	10.9	185
9	30.0	9.8	204
10	28.3	9.6	220
Average	31.0	9.7	205



it was not possible to correlate heat transfer coefficients obtained in a series of tests with syrup of various solids concentrations over a range of temperature differences while operating the unit at total reflux.

Although rapid fouling occurred during the reflux tests, there was no evidence of it during actual processing runs. Table V presents the heat transfer coefficients for each eight hour shift of the run for which material balances were given in Table III. The nominal retention time of syrup in the evaporator was 20 hr. Except for a possible slight decrease in the first three shifts, there was no downward trend in the heat transfer coefficient. All other runs gave similar results.

Average heat transfer coefficients obtained during evaporation tests are arranged in order of increasing temperature difference in Table VI. All values are averages taken over periods of at least 24 hr. with the rate of steam condensate determined every two hours.

TABLE VI  
EVAPORATOR HEAT TRANSFER COEFFICIENTS TO BOILING SYRUP  
DURING PROCESSING TESTS

Solids in syrup, %	Temp. diff., ° F.	Evaporator liquid level, inches above tube sheet	Coefficient, B.t.u. per (hr.) (sq. ft.) (° F.)
31.0	9.7	30	205
31.0	13.5	30	133
46.9	13.5	30	149
42.1	14.0	30	137
28.1	23.0	40	241
41.2	29.0	30	85

#### DIOL PURIFICATION

Butanediol was separated from fermented mash solids by a stripping-scrubbing operation described elsewhere (6) to yield an aqueous solution containing 20–25% diol. This scrubbing column product also contained fairly high concentrations of acid impurities, both free and combined, and smaller amounts of combined ammonia. Sodium hydroxide was added to prevent distillation of impurities, but an excess would be undesirable as it would combine with the diol. Operational methods, material balances, and heat transfer data for one method of recovering diol from scrubber product are given in the following sections.

##### *Concentration of Scrubber Product*

After mixing sodium hydroxide with the scrubber product, 6–7 gal. of the mixture was pumped to the evaporator. Steam was automatically controlled by the column pressure drop and vapor from the evaporator was admitted to the mid-point of the column. Any water reaching the reboiler was vaporized by maintaining a steam pressure of 10 p.s.i. in the calandria. The distillate take-off rate was adjusted to give a reflux ratio (reflux/take-off) of about

one-third. Feed was added with the centrifugal pump and controlled by evaporator liquid level. As the material in the evaporator became more concentrated, the vapor became richer in diol, which collected in the reboiler. After about eight hours, the desired concentration was reached in the evaporator and the operation was changed to allow withdrawal of the solids resulting from the addition of sodium hydroxide. Feed was then added with the piston pump and the centrifugal pump used to remove the residue at a rate controlled by the evaporator level. Steam to the evaporator was controlled by the pressure, the control point being adjusted to give the correct residue rate. Steam to the reboiler was controlled by the pressure drop. The removal of crude diol was controlled by the liquid level in the reboiler and take-off was regulated to give a diol concentration of about 50% in the crude. Since the boiling point of the water-diols system begins to increase rapidly at about 50%, variation in concentration could be quickly detected by a change in the temperature at the bottom of the column. This method of operation gave good control of all flow rates and concentrations.

The material balance for 48-hr. operation during one run is given in Table VII and heat transfer data are given in Table VIII.

TABLE VII  
MATERIAL BALANCE FOR CONCENTRATION OF SCRUBBER PRODUCT  
(Period of operation: 48 hr.)

	Total material	Diol	Solids
Feed, lb.	1103.3	181.93	27.47
Distillate, lb.	737.1	1.35	0.00
Crude diol, lb.	212.0	105.13	0.00
Residue, lb.	117.2	79.88	23.38
Out, lb.	1066.3	196.36	23.38
Out, % of feed	96.65	102.43	86.11
Loss, lb.	37.0	-4.43	4.09

TABLE VIII  
HEAT TRANSFER DATA FOR CONCENTRATION OF SCRUBBER PRODUCT

	Evaporator	Reboiler
Steam pressure, p.s.i.	18.8	9.0
Vapor temperature, ° F.	228.4	216.7
Temperature difference, ° F.	28.3	20.2
Coefficient, B.t.u. per (hr.) (sq. ft.) (° F.)	39	169
Diol, %	68.16	49.6
Solids, %	19.95	0.0
Evaporator liquid level, inches above bottom tube sheet	44	

#### *Rectification of Crude Diol*

The crude diol was separated into diol and water fractions by continuous vacuum distillation in the column. Crude diol was pumped with the piston

pump to the mid-point of the column, withdrawal of impure diol was regulated by the liquid level, and the distillate rate was manually adjusted to the proper proportion of the feed. Steam to the reboiler was controlled by pressure drop until the diol concentration was high enough to allow control by the bottom temperature. The column pressure drop could then be adjusted by changing the feed rate. Material balances for this operation are given in Table IX.

TABLE IX  
MATERIAL BALANCE FOR RECTIFICATION OF CRUDE DIOL

	Total material	Diol
Feed, lb.	292.8	141.8
Distillate, lb.	137.0	1.6
Product, lb.	158.2	147.5
Out, lb.	295.2	149.1
Out, % of feed	100.82	105.10
Loss, lb.	-2.4	-7.2

Other data are:

Liquid to vapor ratio at top of column	0.154
Temperature at top of column, ° F.	129.2
Reboiler steam pressure, p.s.i.	45
Vapor temperature, ° F.	269.0
Temperature difference, ° F.	23.7
Heat transfer coefficient, B.t.u. per (hr.) (sq. ft.) (° F.)	66

### *Final Distillation*

As a final purification step, the impure diol was treated with sodium hydroxide and vacuum distilled from the reboiler of the column. Feed was drawn into the column by vacuum and was regulated by hand. Steam was controlled by pressure drop and no reflux was used. At the end of the run the residue in the reboiler was washed out with a known amount of water. No heat transfer or flow rate data were recorded.

### DISCUSSION

During investigations of recovery of diol from fermented molasses mashes, this equipment was in almost constant use for over 18 months. For all operations, the capacities of the evaporator, reboiler, and condenser were adequate to operate the column at full capacity. The heat loss from the column was relatively large and made it impracticable to calculate transfer unit heights except when operating at total reflux. Insulation of exposed flanges would not have reduced the variation in liquid and vapor rates throughout the packing since the packed section of the column contained no flanges. The vapor head and separator effectively prevented carry-over of solids by foam or entrain-

ment, but very little foam was formed in the evaporator and it was never necessary to add antifoam agents. In a standard vertical tube evaporator, foaming of fermented molasses mashes was a major problem and it was necessary to add an antifoam agent continually. The installation of a flexible automatic control system has been justified. Without it, smooth continuous operation would not have been possible.

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# THE EFFECT OF MOISTURE ON THE THERMAL PROPERTIES OF WHEAT<sup>1</sup>

BY IRENE MOOTE

## ABSTRACT

The thermal properties of wheat containing various amounts of moisture, from 1.4% to 15.9% of the dry weight, were determined by placing the wheat in a cylindrical apparatus with the heating element stretched along the central axis and by measuring the radial temperatures. Three general conclusions can be drawn from the results of the experiments. The properties of dry wheat are: thermal conductivity 0.00030 cal. per sq. cm. per sec. per °C./cm.; thermal diffusivity 0.0012 sq. cm. per sec., specific heat 0.31 cal. per gm. per °C.; all three properties vary slightly depending on the kind of wheat. An increase in the moisture content of the wheat causes a linear increase in the thermal conductivity, a slight decrease in the thermal diffusivity and an increase in the specific heat. The migration of moisture through the wheat under the influence of a temperature gradient causes the thermal properties to vary at different locations in the wheat, depending on the moisture content.

## INTRODUCTION

In a paper published recently Babbitt (2) measured the thermal properties of wheat by means of a cylindrical apparatus with an axial heating element. The wheat used in his experiment contained 9.2% by weight of moisture. To derive the specific heat of the wheat alone, it was necessary to allow for the heat capacity of the adsorbed water. In his paper Babbitt assumes the adsorbed water contributes to the specific heat as if it were liquid water. This may not be so, since the adsorbed molecules of water do not have the same freedom of movement as liquid molecules. To dispel this uncertainty it seemed worthwhile to make a series of measurements which would give some measure of the specific heat of the adsorbed water itself.

Thus it was decided to measure, following the procedure used by Babbitt, the thermal properties of a sample of wheat at a number of different moisture contents, beginning with dry wheat and increasing the moisture content for each consecutive measurement. However, owing to the temperature gradient, the moisture in the wheat migrated from points of high to points of low temperature and the latent heat exchanges involved upset the measurement of the thermal properties. It was known that there is a tendency for moisture to migrate when there is a temperature gradient in wheat, but previous measurements (Anderson, Babbitt, and Meredith (1) and Babbitt (3)) had indicated that this migration took place very slowly and it seemed unlikely that it should nullify our measurements. It is evident from the results following that this migration is sufficient, even at low moisture contents, to affect the measurements and that it is impossible to obtain from them an estimate of the contribution to the heat capacity made by the adsorbed water.

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However these measurements do give valuable information on the migration of water vapor through granular beds under a temperature gradient and show how this migration affects the thermal properties of the material. The effect of migration of moisture under temperature gradients is important in thermal insulating materials, and in soils. Therefore it seemed worthwhile to report our results and conclusions on the behavior of water vapor in wheat under the influence of a temperature gradient.

#### APPARATUS AND METHODS

The apparatus for measuring the thermal properties of wheat was the one used and described by Babbitt (2); Figs. 1, A and B are photographs of a second apparatus built and used for some of the experiments. The wheat container was a galvanized iron cylinder 1 ft. in diameter and 2 ft. in length, closed with bakelite ends. The heating element was a single strand of 26 gauge Chromel A wire stretched along the axis of the cylinder. The 12 thermocouples were placed along a diameter halfway down the cylinder at radii of 0.5, 1.5, 2.5, 3.5, 4.5, and 5.5 in. The cylinder was placed in an insulated box fitted with a fan for circulating the air and with a heater and thermostat for controlling the temperature of the air surrounding the cylinder. Fig. 1, A and B also show the thermocouple switch, ice-bath reference junction, and the control panel for the heating circuit.

Two lots of wheat were used in the tests. Both lots were No. 1 Northern Manitoba grade, but one lot was obtained in 1949 and the other in 1950. The first lot was dried in a vacuum oven at 105° C. to a moisture content of 1.4% dry weight before test. Before each succeeding test, water was added to the wheat and the whole was mixed well and allowed to stand for a couple of days until adsorption of the water was complete. By this method the moisture content of the wheat was increased by steps to 7.9%. The second lot of wheat contained 15.9% by weight of moisture when first used. It was tested in this state and then before each succeeding test it was partially dried in an oven, its moisture content being decreased by steps to 5.6%.

The moisture content of the wheat was determined by an oven method. Five samples of wheat were taken from the cylinder while it was being filled. These were weighed, then dried in a vacuum oven at 105° C. to constant weight (i.e. for a week or 10 days). The moisture content was calculated for each sample from its weight before and after drying. The average of these five values was used as the moisture content of the wheat being tested.

After the temperatures of the wheat had come to equilibrium with the air in the box surrounding the cylinder, the heating element was switched on. The temperatures of the wheat at the 12 thermocouples were taken at intervals until a state of temperature equilibrium was reached. The average temperatures at the six radii were calculated and then reduced by subtracting from them the initial temperature of the wheat. This temperature, also called the zero temperature, was the same as that of the air surrounding the cylinder, and was approximately 90° F. for some of the tests and approximately 97° F.



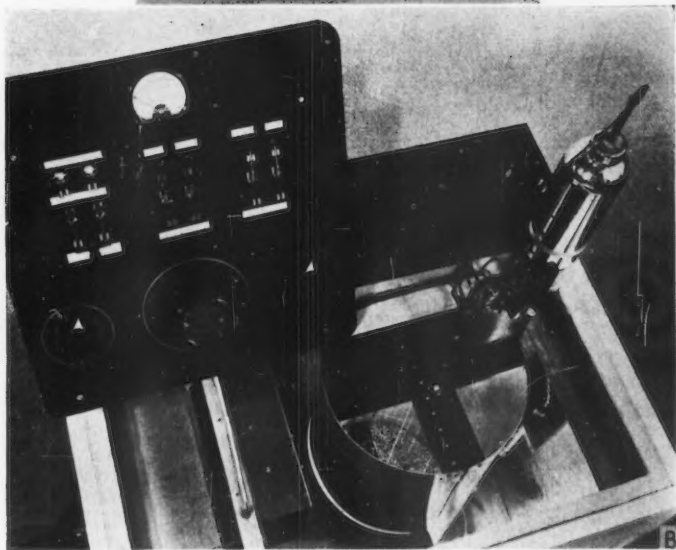
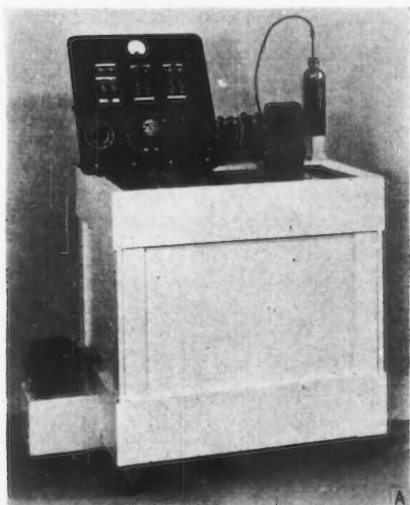


FIG. 1A. A side view of the apparatus showing the insulated box, the motor for the fan, the thermocouple switch on top of the cylinder, the control panel for the heating circuit, and the thermos flask containing the ice bath and reference junction.

B. A top view of the apparatus showing the heater at one end of the insulated box, the thermostat at the other end, and the baffles for controlling the air flow, as well as the cylinder containing the heating element, thermocouples, and a small amount of wheat. The control panel, thermocouple switch, and thermos flask are also shown.

for the others. The reduced temperatures at each radius were plotted against the time of heating, and these curves, which give the rate of change in temperature, were used in calculating the thermal diffusivity of the wheat. The final reduced temperature distribution at the six radii was used in calculating the thermal conductivity of the wheat.

#### THEORETICAL CONSIDERATIONS

The Fourier heat equation for the temperature at any point in a right circular cylinder whose initial temperature is a function of the radius only is

$$[1] \quad \frac{dv}{dt} = \frac{k}{c\rho} \left( \frac{d^2v}{dr^2} + \frac{1}{r} \frac{dv}{dr} \right)$$

where  $v$  is the temperature,  $t$  is the time,  $r$  is the radius,  $k$  is the thermal conductivity,  $c$  is the specific heat, and  $\rho$  is the density. According to Carslaw (4), in a circular cylinder where the initial temperature is constant, taken as zero, and heat is supplied at a constant rate along the axis of the cylinder, for steady state conditions the equation for heat flow is

$$[2] \quad V_r = \frac{I^2 R}{2\pi k} \log_e a - \frac{I^2 R}{2\pi k} \log_e r$$

where  $V_r$  is the temperature at radius  $r$ ,  $I$  is the current, and  $R$  is the resistance per unit length of the heating element,  $a$  is the radius corresponding to boundary temperature conditions, and  $k$  is the thermal conductivity. To determine the thermal conductivity  $k$ , equation [2] is compared with

$$[3] \quad V = B + C \log_e r$$

obtained from the final temperature distribution of the experiment.

$$\text{Thus } B = \frac{I^2 R}{2\pi k} \log_e a$$

$$\text{and } C = -\frac{I^2 R}{2\pi k}$$

and since  $B$ ,  $C$ , and  $a$  can be obtained from the slope and intercepts of the equilibrium curve,  $k$  is calculated from

$$[4] \quad \frac{I^2 R}{2\pi B} \log_e a$$

or

$$[5] \quad -\frac{I^2 R}{2\pi C}$$

The thermal diffusivity is defined as the constant in the Fourier equation [1] i.e.

$$[6] \quad \kappa = \frac{k}{c\rho}$$

Carslaw (4) gives the following solution of the Fourier equation when the initial temperature is a function of the radius only and when the surface of the cylinder is kept at zero temperature

$$[7] \quad v = \sum_{n=1}^{\infty} A_n J_0(\alpha_n r) e^{-\kappa \alpha_n^2 t}$$

where  $v$  is the temperature at time  $t$ ,  $A_n$  is a constant determined from the initial temperature distribution,  $J_0(\alpha_n r)$  is Bessel function of order zero of the first kind, and  $\alpha_n$  are the roots of  $J_0(\alpha_n a) = 0$ ,  $\kappa$  is the thermal diffusivity. From this Babbitt (2) has derived the equation for an experiment in which the surface of the cylinder is maintained at the initial or zero temperature of the whole mass and the final temperature is a function of the radius given by

$$V = B + C \log_e r$$

as

$$[8] \quad v = V_r - \sum_{n=1}^{\infty} A_n J_0(\alpha_n r) e^{-\kappa \alpha_n^2 t}$$

where

$$[9] \quad A_n = \frac{B \frac{a}{\alpha_n} J_1(\alpha_n a) + C \left[ \frac{a}{\alpha_n} J_1(\alpha_n a) \log_e a - \frac{1}{\alpha_n^2} \right]}{a^2 / 2 J_1(\alpha_n a)^2}$$

and  $V_r$  is the final temperature. To calculate  $\kappa$  one experimental point, chosen so that only one term of equation [8] is significant, is substituted into equation [8] after  $B$ ,  $C$ ,  $a$ ,  $\alpha_n$  and  $A_n$  have been calculated.

After the values of thermal conductivity  $k$  and thermal diffusivity  $\kappa$  have been calculated, the specific heat is calculated using equation [6].

This theory deals with the conduction of heat in a cylinder when the initial, final, and boundary conditions are given, but does not indicate the effects of temperature changes from other sources. It has been noted that during the time of an experiment moisture migrates through the wheat and this affects the thermal properties. This movement of moisture under the influence of temperature gradients has been described by Oxley (5) and by Anderson, Babbitt, and Meredith (1). If there were no movement of moisture or change in the thermal properties during an experiment equilibrium should be reached in 80 to 100 hr. For this reason it was decided to calculate the thermal properties from the temperatures of the wheat after 100 hr. of heating and to compare these values with those determined from the final temperatures. While the former do not represent the true thermal properties at the initial moisture content because of the moisture migration, they should be a better approximation to them than those obtained from the final temperatures.

#### EXPERIMENTAL RESULTS

The thermal conductivity was calculated from the slope of the curve obtained when the final temperatures, given in Table I, were plotted against the logarithm of the radius. These equilibrium curves were drawn for each test and two of them, namely those for wheat containing 2.2% and 15.9%

TABLE I  
FINAL EQUILIBRIUM TEMPERATURE DISTRIBUTION OF WHEAT

Moisture content, % dry weight	Radius in inches					
	0.5	1.5	2.5	3.5	4.5	5.5
	Final temperatures in ° F.					
1.4	48.0	27.6	17.9	11.5	6.6	2.8
2.2	47.8	27.3	17.6	11.2	6.5	2.7
2.6	46.1	26.3	16.8	10.8	6.4	2.8
3.0	47.4	26.8	17.3	11.0	6.4	2.7
3.6	46.5	26.4	16.8	10.6	6.1	2.6
4.8	46.1	26.1	16.9	10.8	6.3	2.7
5.6	42.9	24.4	15.7	10.2	5.9	2.6
6.9	45.2	25.4	16.4	10.5	6.2	2.7
7.4	43.5	24.7	15.9	10.3	6.1	2.6
7.4	43.2	24.1	15.4	9.9	5.6	2.3
7.9	44.7	25.2	16.3	10.5	6.2	2.7
8.3	41.8	23.3	14.9	9.5	5.5	2.4
10.8	41.8	23.2	14.9	9.6	5.5	2.3
12.2	41.8	23.5	15.2	9.8	5.7	2.4
15.9	42.0	23.5	15.2	9.7	5.7	2.6

moisture, are shown in Fig. 2. The equilibrium curve for wheat containing 2.2% moisture is a straight line of the form  $V = B + C \log_e r$  where  $V$  is the final temperature at radius  $r$  and  $B$  and  $C$  are constants; whereas the equilibrium curve for wheat containing 15.9% moisture is not a straight line; its slope gradually increases at the smaller radii. This deviation from a straight line was noted in the equilibrium curves for wheat containing more than 3.5% moisture, and is illustrated in Fig. 3 where the slopes of the equilibrium curves have been plotted against the radius for five of the tests. Fig. 3 shows that,

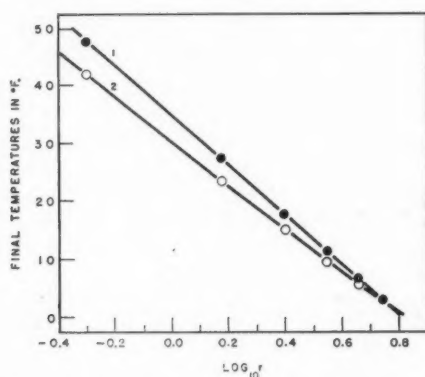


FIG. 2. Final temperature distribution of wheat containing (1) 2.2% and (2) 15.9% by weight of moisture.

as the moisture content of the wheat is increased, the amount of deviation from the straight line becomes greater and the deviation begins at larger radii i.e. farther from the central heating axis. There is a tendency for the slopes at all moisture contents to approach that at moisture content of 2.2% as radius becomes smaller. Since the thermal conductivity is inversely proportional to the slope of the equilibrium curve, the graphs of Fig. 3 indicate

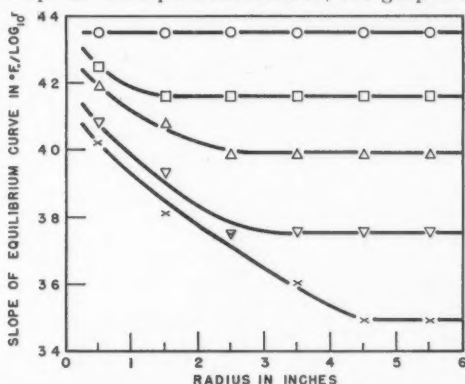


FIG. 3. The slope of the equilibrium curve plotted against the radius for wheat containing 2.2%, 4.8%, 6.9%, 7.4%, and 15.9% of moisture respectively.

that the thermal conductivity of wheat containing more than 3.5% moisture must be less near the central heating axis than it is near the outside of the cylinder. The difference between the values of conductivity at these two positions is greater at higher moisture contents of the wheat. As there may have been a transfer of moisture through the wheat during the experiments owing to the temperature gradient, it was decided to measure the moisture content of the wheat near the center and also near the edge of the cylinder at the end of those experiments in which the initial moisture content was more than 4%. Table II contains the results of these measurements and shows very clearly that a considerable amount of moisture has been transferred during the experiments.

TABLE II  
CHANGE IN MOISTURE CONTENT OF WHEAT DURING TEST, % DRY WEIGHT

Moisture content before test	Moisture content after test	
	Near axis	Near the outside of the cylinder
4.8	2.9	4.9
5.6	3.7	5.9
7.4	4.2	7.9
7.9	5.1	8.6
8.3	5.0	8.7
10.8	5.8	11.8
12.2	5.7	13.2
15.9	8.1	17.2

## CALCULATION OF CONDUCTIVITY, DIFFUSIVITY, AND SPECIFIC HEAT

The final temperature distribution curves similar to those in Fig. 2 were drawn for each test on the wheat and from them the values of  $B$ ,  $C$ , and  $a$  were calculated. In the tests on wheat containing appreciable amounts of moisture, where the equilibrium curves are not straight lines, the straight portions drawn through the points corresponding to the larger radii were used. The thermal conductivity for each test was then calculated from equation [4] or [5].

For reasons discussed above, these calculations were repeated using the temperatures of the wheat after 100 hr. of heating instead of the final temperatures in drawing the equilibrium curves. These latter values of conductivity are called the  $k'$  values and are given in Table III as well as the  $k$  values. In

TABLE III  
THERMAL CONDUCTIVITY RESULTS FOR WHEAT AT VARIOUS MOISTURE CONTENTS

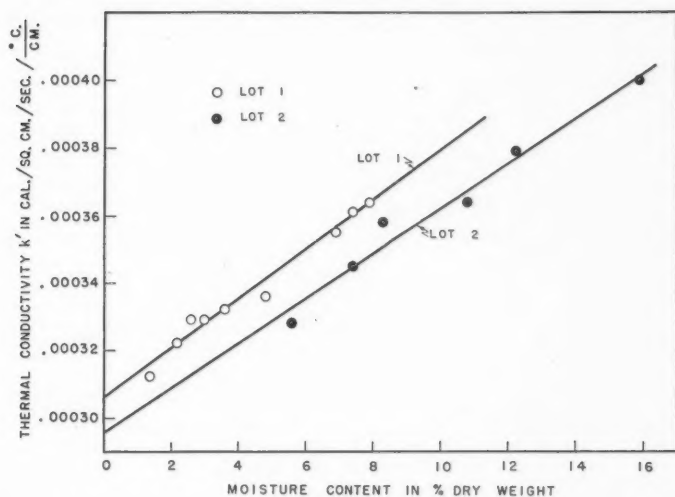
Wheat lot No.	Moisture content, % dry weight	Thermal conductivity $k$ , cal./sq. cm./sec./ $\frac{^{\circ}\text{C.}}{\text{cm.}}$	Thermal conductivity $k'$ , cal./sq. cm./sec./ $\frac{^{\circ}\text{C.}}{\text{cm.}}$
1	1.4	.000313	.000312
1	2.2	.000316	.000324
1	2.6	.000332	.000329
1	3.0	.000324	.000329
1	3.6	.000335	.000332
1	4.8	.000328	.000336
2	5.6	.000325	.000328
1	6.9	.000341	.000355
1	7.4	.000352	.000361
2	7.4	.000331	.000345
1	7.9	.000342	.000364
2	8.3	.000347	.000358
2	10.8	.000351	.000364
2	12.2	.000343	.000379
2	15.9	.000367	.000400

Fig. 4 the  $k'$  values of thermal conductivity are plotted against the moisture content of the wheat. Table III and Fig. 4 indicate that the thermal conductivity of dry wheat is approximately 0.00030 cal. per sq. cm. per sec. per  $^{\circ}\text{C./cm.}$  and varies slightly for the two wheats measured and that it increases with moisture content at a rate of approximately 0.000007 cal. per sq. cm. per sec. per  $^{\circ}\text{C./cm.}$  for every 1% dry weight increase in moisture content. There is a considerable difference between the  $k$  values and  $k'$  values of the thermal conductivity of wheat when it contains more than 4.8% of the dry weight of moisture.

For the calculation of thermal diffusivity  $\kappa$ , the values of  $A_1$ ,  $A_2$ , and  $A_3$  were first calculated using equation [9]. Then one point was chosen from the experimental time-temperature curves where  $t$  was sufficiently large that only one term of equation [8] was significant, and these values for  $v$  and  $t$  were substituted into equation [8]

$$v = V_r - \sum_{n=1}^{\infty} A_n J_0(\alpha_n r) e^{-\alpha_n^2 \kappa t}$$



FIG. 4. Variation of the thermal conductivity  $k'$  with the moisture content of the wheat.

The results of these calculations of the thermal diffusivity  $\kappa$  for the experiments are given in Table IV. The thermal diffusivity was also calculated using the temperatures after 100 hr. of heating in place of the final temperatures  $T_r$ ,

TABLE IV

THERMAL DIFFUSIVITY RESULTS OF WHEAT AT VARIOUS MOISTURE CONTENTS

Wheat lot No.	Moisture content, % dry weight	Thermal diffusivity $\kappa$ , sq. cm. per sec.	Thermal diffusivity $\kappa'$ , sq. cm. per sec.
1	1.4	.00113	.00115
1	2.2	.00105	.00113
1	2.6	.00114	.00116
1	3.0	.00113	.00114
1	3.6	.00107	.00110
1	4.8	.00094	.00105
2	5.6	.00109	.00111
1	6.9	.00101	.00111
1	7.4	.00094	.00107
2	7.4	.00096	.00106
1	7.9	.00089	.00105
2	8.3	.00095	.00105
2	10.8	.00089	.00102
2	12.2	.00080	.00105
2	15.9	.00072	.00106

and these results called  $\kappa'$  are also given in Table IV. The following conclusions were drawn:

1. The thermal diffusivity of dry wheat is approximately 0.00117 sq. cm. per sec. and it decreases as the moisture content is increased.

2. The rate of decrease of the thermal diffusivity is greater for the  $\kappa$  values than for the  $\kappa'$  values.

The specific heat was calculated from the corresponding values of  $k$  and  $\kappa$  and the density  $\rho$  using equation [6]

$$c = \frac{k}{\kappa \rho}.$$

The density  $\rho$  was the bulk density of the wheat and was measured for each experiment. In Table V the values of the density as well as the calculated

TABLE V  
CALCULATED VALUES OF SPECIFIC HEAT OF WHEAT AT VARIOUS MOISTURE CONTENTS

Wheat lot No.	Moisture content, % dry weight	Moisture content, % wet weight	Density, gm. per cc.	Specific heat, $c' = \frac{k'}{\kappa' \rho}$
1	1.4	1.4	0.842	0.319
1	2.2	2.2	0.846	0.339
1	2.6	2.5	0.825	0.344
1	3.0	2.9	0.822	0.351
1	3.6	3.5	0.853	0.354
1	4.8	4.5	0.859	0.373
2	5.6	5.3	0.828	0.357
1	6.9	6.4	0.858	0.373
1	7.4	6.9	0.881	0.385
2	7.4	6.9	0.831	0.392
1	7.9	7.4	0.860	0.403
2	8.3	7.7	0.828	0.412
2	10.8	9.7	0.831	0.429
2	12.2	10.9	0.821	0.440
2	15.9	13.7	0.821	0.460

values of specific heat are given for the various moisture contents of the wheat. The values of specific heat were calculated from the  $k'$  and  $\kappa'$  values of conductivity and diffusivity as these values were not affected by the migration of moisture to the same extent as the  $k$  and  $\kappa$  values. At very low moisture contents, there was no significant movement of moisture so the values for specific heat of wheat containing 1.4% and 2.2% moisture are presumably correct. By extrapolation from these values, the specific heat of dry wheat is found to be 0.314 and 0.310 for the two lots of wheat.

Pfalzner (6) has determined the specific heat of wheat at various moisture contents by a method of mixtures. He obtained the following results for three lots of wheat:  $c = .283 - .00724U$ ;  $c = .301 - .00733U$ ; and  $c = .288 - .00828U$  respectively, where  $c$  is the specific heat and  $U$  is the moisture content expressed in per cent of the wet weight of the wheat. His values of specific heat of dry wheat (.283 to .301) agree fairly well with our values (.310 and .314). In Fig. 5 are plotted the values of specific heat given in Table IV as well as the values obtained by using our values of the specific heat of dry wheat and the proportionality constants from Pfalzner.

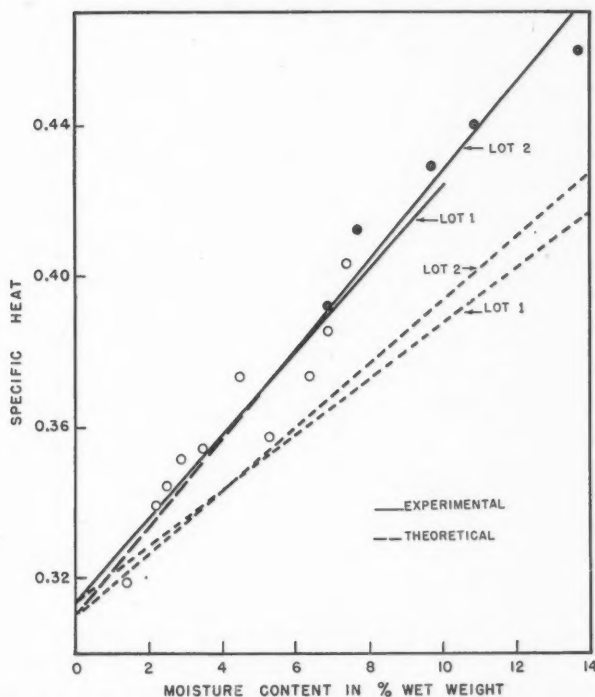


FIG. 5. Variation of the specific heat with the moisture content of the wheat. — Specific heat values obtained from the experimental results. - - - Specific heat values obtained from Pfalzner's equations.

#### GENERAL DISCUSSION

In these experiments the effect of moisture on the thermal properties was twofold; one effect was due to the presence of moisture in the wheat kernels and the other was due to the migration of moisture from the hot to the cooler wheat, and these two effects could not be separated. The migration of moisture through the wheat took place by diffusion of the water vapor through the intergranular air spaces and by the interchange of moisture between the wheat kernels and the air surrounding them. Of these two processes the adsorption and desorption of water vapor by the wheat would have the greater effect on the thermal properties. The heat of desorption required when the hot wheat gave up moisture and the heat of adsorption acquired when the cool wheat adsorbed this moisture resulted in a heat transfer from the central axis toward the outside of the cylinder. This transfer of heat increased the rate of heat flow causing an apparent increase in the thermal conductivity and thermal diffusivity. But when equilibrium had been reached, this transfer of heat no longer took place and the measured values of thermal conductivity and thermal diffusivity were less than they were while migration was taking place.

This is illustrated by the results in Tables III and IV; for the  $k'$  and  $\kappa'$  values of thermal conductivity and thermal diffusivity respectively obtained during the migration of moisture are greater than the corresponding  $k$  and  $\kappa$  values obtained after migration. After equilibrium had been reached, there was a gradient in moisture content across the wheat. Thus the thermal conductivity varied across a radius of the cylinder, being least near the axis and greatest near the outside as indicated by the graphs in Fig. 3.

The time required for the temperature of the wheat to reach equilibrium depended on the moisture content. At low moisture contents the equilibrium temperatures were reached in less than one week; at higher moisture contents the time required was much longer—in fact up to 400 hr. for the wheat containing 15.9% moisture. This may be explained by the movement of moisture through the wheat and its effect on the thermal conductivity. For as the moisture moved through the wheat under the influence of the temperature gradient, the thermal conductivity of the wheat near the central axis decreased and its temperature rose. The rise in temperature increased the temperature gradient across the wheat and more moisture began to diffuse through the wheat. This process continued until a state of moisture equilibrium as well as temperature equilibrium had been established. Whether this state of equilibrium had been reached in the time of an experiment is difficult to say. The experiments were considered completed when there was no longer an appreciable change in temperature over a period of a day—but this may have been only a gradual deceleration of the process, not its conclusion.

The graphs in Fig. 3 show how the migration of moisture affects the thermal conductivity measurements. The thermal conductivity varies within the wheat depending on its moisture content, but the measurements are based on a constant value of conductivity throughout the bulk. The difference between the  $k$  values and the  $k'$  values of thermal conductivity shown in Table III indicates that the movement of moisture in the wheat causes the measured values of thermal conductivity to be lower than the actual values if no movement had taken place.

The thermal diffusivity measurements are also affected by the migration of moisture. Table IV shows that the  $\kappa$  values decrease at a greater rate than the  $\kappa'$  values of diffusivity when the moisture content of the wheat is increased. Since the  $\kappa$  values are affected by the migration of moisture more than the  $\kappa'$  values, this indicates that the migration of moisture causes the measured values of thermal diffusivity to be lower than the actual values if no migration had taken place.

The specific heat results are also affected by the migration of moisture since they depend on  $k$  and  $\kappa$ . Using the  $k'$  values of conductivity and the  $\kappa'$  values of diffusivity in calculating the specific heat, this effect of migration will be less. However after comparison of these calculated values of specific heat with those using Pfalzner's relation between specific heat and moisture content in Fig. 5, it appears that our values of specific heat are still considerably affected by the migration of moisture.

As mentioned above, there is a slight difference in the thermal properties of the two lots of wheat used. This may be partly due to the difference in the procedure of changing the moisture content. The first lot of wheat was dried and moisture was gradually added to it, whereas the second lot was dried by steps and tests made between the drying periods. After the wheat had been completely dried, its ability to absorb moisture may have been changed.

#### ACKNOWLEDGMENT

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## NOTE

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### A NOTE ON THE PRODUCTION OF 2,3-BUTANEDIOL FROM GLUCOSE BY STRAINS OF *BACILLUS CEREUS*\*

BY FLORENCE R. TAMBOLINE

As part of a survey of Voges-Proskauer positive species of the genus *Bacillus* for the production of 2,3-butanediol, a study was made of 46 strains of *Bacillus cereus*. Nine strains were obtained from N. R. Smith, U.S. Bureau of Plant Industry Station, Beltsville, Md., and the rest were isolated locally.

All cultures gave a positive V-P reaction by Smith's method (2) and all fermented glucose in his medium (2) although three to seven days were required for the detection of significant amounts of acid.

Fermentations were carried out in 125-ml. filtering flasks containing 50-ml. quantities of the following medium: glucose, 5.0%; yeast extract, 0.5%; potassium dihydrogen phosphate, 0.05%; dipotassium hydrogen phosphate, 0.05%; magnesium sulphate heptahydrate, 0.02%; calcium carbonate, 2.0%. The calcium carbonate was sterilized in the fermentation flasks for four hours in an oven at 175°C. The other components of the medium were autoclaved at 121°C. for 15 min. in four separate portions (the phosphate salts together) to give the desired concentrations when added aseptically to the fermentation flasks.

Cultures for inoculation were grown in 10-ml. quantities of a 1.0% glucose - 0.5% yeast extract broth in 50-ml. Erlenmeyer flasks for 18 hr. at 30°C. on a shaker.

After addition of a 6% inoculum each fermentation flask was fitted with a sterile rubber stopper through which passed a glass tube with one end tapered to a small opening immersed well below the surface of the culture. Through this tube water-saturated commercial nitrogen (not freed of oxygen) was passed into the culture at a rate that maintained active bubbling throughout the fermentation. All cultures were incubated at 30°C. on a rotary shaker at a speed of 125 r.p.m.

After 10 days' incubation the cultures were examined for purity, cleared of bacteria and protein with zinc hydroxide (3), and analyzed for residual glucose by the method of Underkofler *et al.* (4) and for 2,3-butanediol by the direct method of Leslie and Castagne (1).

The extent of glucose dissimilation ranged from 63 to 98% with about three-quarters of the strains showing 80%, or more, dissimilation (Table I). Diol production varied from 0.20 to 1.34%. Only seven cultures produced more than 1.0% diol and 17 gave less than 0.5%.

Three strains were selected for further study to determine whether the diol yields could be increased by altering fermentation conditions. Two sources of inoculum were employed in this study: the broth cultures described previously

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TABLE I

DISTRIBUTION OF 46 STRAINS OF *Bacillus cereus* ON THE BASIS OF GLUCOSE DISSIMILATION AND 2,3-BUTANEDIOL PRODUCTION\*

Diol formed, %	Glucose dissimilated, %			
	61-70	71-80	81-90	91-100
0.0 - 0.5	-	7	9	3
0.5 - 1.0	-	2	8	10
> 1.0	1	-	3	3

\*Conditions: Cultures incubated under nitrogen on a shaker at 125 r.p.m. for 10 days at 30°C.

and suspensions in normal saline solution prepared from 18-hr. nutrient agar slants. Both types of inoculum contained approximately the same number of cells. Nitrogen was bubbled through half of the cultures during incubation as in the previous experiment. The other half of the cultures were incubated aerobically in 125-ml. Erlenmeyer flasks fitted with cotton plugs. Residual glucose and diol were estimated after 2, 4, 6, and 10 days' incubation.

None of the conditions tested led to significant variation in diol production or in glucose dissimilation. In all cultures dissimilation of glucose was slow with 38 to 67% dissimilation in 48 hr. and 67 to 97% in 10 days.

The results of this study indicate that *B. cereus* dissimilates glucose too slowly and produces too little diol to be considered as a potential organism for the industrial production of 2,3-butanediol.

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